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# AMRF COMPOSITES FABRICATION WORKSTATION<sup>1</sup>

## A Test Methodology to Measure the Quality of Thermoplastic Composite Parts

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### ABSTRACT

The Robot Systems Division of the National Institute of Standards and Technology (NIST) requires a test methodology to measure the quality of the composite parts produced by an advanced manufacturing workstation being assembled in the Automated Manufacturing Research Facility (AMRF). The workstation will be used to study methods of fabricating complex shaped, continuous carbon fiber reinforced, thermoplastic composite parts using pre-impregnated tow. Fabrication will be accomplished using in-situ consolidation and tow placement techniques via two cooperating robot manipulators. The test methodology will utilize a combination of material testing techniques to measure the consolidation of fabricated parts during implementation of the manufacturing process. This paper discusses the mechanisms of thermoplastic consolidation and the various defects associated with poor consolidation. Several testing techniques are discussed concerning their ability to locate, identify and/or quantify these defects. This discussion is followed by a trade off analysis of all considered testing techniques in an attempt to determine the most effective test methodology to measure the quality of thermoplastic composite parts.

### INTRODUCTION

The Automated Manufacturing Research Facility (AMRF) is a testbed at the National Institute of Standards and Technology (NIST) in which several factory automation issues are being addressed. The AMRF consists of several workstations which are used to study machining, deburring, inspection and handling of metal parts, as well as the associated control, data processing, machine programming and scheduling issues. A composites workstation [1] is being developed and incorporated into the AMRF in order to apply AMRF principles to the fabrication of high performance, complex shaped thermoplastic composite parts. These complex parts, which are typically used in the aircraft and defense industry, are now produced with thermoset composites using very time and labor intensive manufacturing techniques.

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Thermoset prepreg processing requires the use of expensive autoclaves and timely cure cycles. Thermosets also have refrigeration and other special handling requirements. Thermoplastics do not require cure processing and therefore can be processed while being applied onto part forms (in-situ consolidation). The capability of thermoplastics to undergo in-situ consolidation eliminates several of the deficiencies associated with thermosets while meeting the performance characteristics of thermosets as a polymer matrix. Thermoplastics have no special storage or handling requirements and unlike thermosets have the ability to be reprocessed. One drawback of thermoplastics is the unavailability of documented methods of in-situ consolidation. These methods must be fully identified through internal efforts and through progress reported from industry efforts.

The AMRF composites workstation will perform in-situ consolidation of continuous carbon fiber reinforced thermoplastic prepreg tow using fiber placement. Fiber placement will be accomplished using two cooperating robot manipulators and a specially designed in-situ consolidation apparatus. The utilization of robot manipulators and the consolidation apparatus will allow the automated production of complex shaped composite parts by introducing heat and pressure to the thermoplastic prepreg tow as it is applied to a part form mandrel.

The composites workstation will consolidate a 6 mm wide prepreg tow of continuous carbon fiber reinforced PEEK thermoplastic, APC-2 produced by ICI Fiberite<sup>2</sup>. Initial efforts will use a two degrees of freedom winder to determine various combinations of temperature, pressure and tow feed rate which result in consolidation of the prepreg tow. Process parameters are shown relative to the contact placement winder in figure 1. These sets of process parameters will then be integrated with various control schemes in attempt to operate the process over a range of feed rates and winding patterns. The final parameters and control schemes, in turn, will be integrated with robot manipulators to develop the AMRF composites workstation.

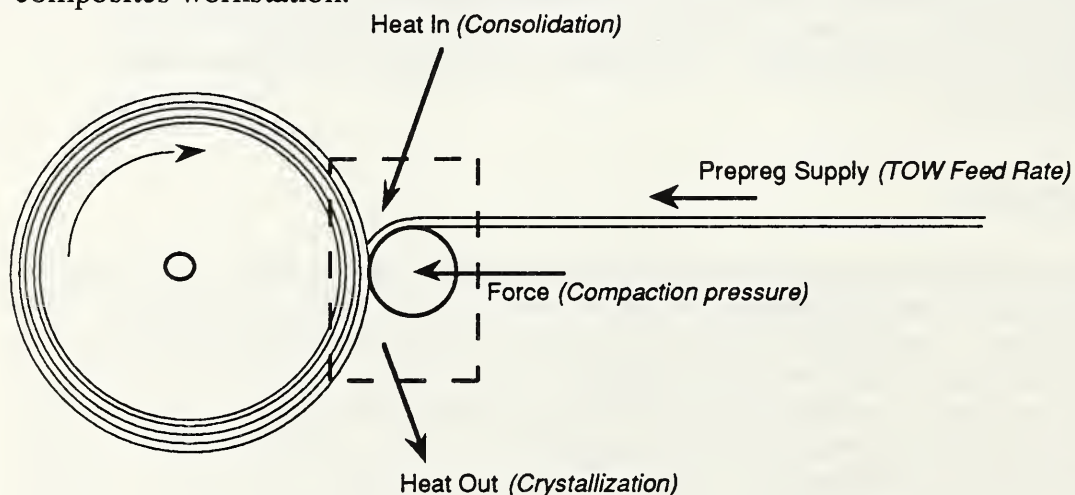


Figure 1 Process Overview

<sup>2</sup> Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Relationships between the heat applied to obtain processing temperature and the tow feed rate will be developed using a thermal model of the hoop winder. Thermal analysis will be accomplished using a finite element analysis package. Relationships developed will be used to approximate initial sets of process parameters. Each set of process parameters will then be evaluated by producing cylindrical parts with the contact placement winder. These parts will undergo inspection and testing as described in the test methodology.

The test methodology will utilize physical, mechanical, and non-destructive test techniques to locate defects at the ply interface. The tests will also provide both an actual view of defects to determine their identity and a quantified assessment of the defects. Based on the elimination of defects, the process parameters will be adjusted until consolidation is achieved. A database of tow feed rates and associated processing temperatures and compaction pressure correlated with consolidation will be maintained. Also, a database of test results will be maintained and used as a quality assurance reference in future composite workstation efforts.

## PROCESSING THERMOPLASTICS

The processing of thermoplastic prepregs can be viewed in two phases, the consolidation [2,3,4] of the plies to one another followed by the crystallization [5,6,7] of the consolidated composite. The level of consolidation achieved while manufacturing thermoplastic composites is dependent on the flow of the thermoplastic matrix during processing. Flow of the thermoplastic is generated through the application of heat and pressure to the moving pre-impregnated tow. The quantity of heat and pressure must be controlled to achieve a good degree of bonding and compaction at a desired tow feed rate. Crystallinity is an ordered arrangement of the molecules that make up a portion of the thermoplastic. The percentage of this crystalline structure affects resulting mechanical properties of the thermoplastic. Crystallization of the thermoplastic occurs during the cooling of the composite immediately following consolidation. The mechanisms of thermoplastic processing (bonding, compaction, and crystallization) are shown in figure 2.

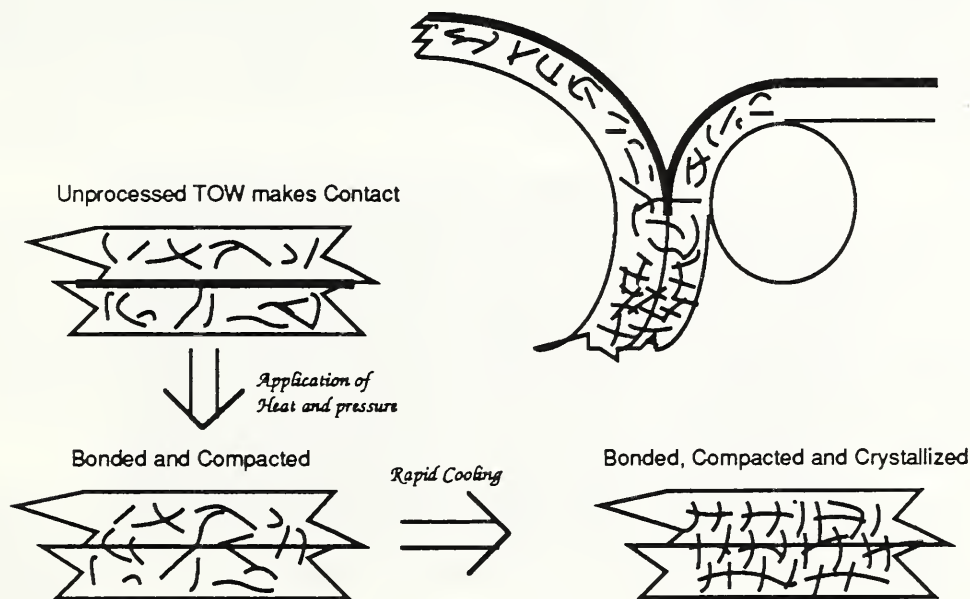


Figure 2 Thermoplastic Processing



The bonding mechanism of processing thermoplastics is a result of autohesion. Autohesion is initiated when the oncoming ply surface and the substrate surface achieve intimate contact. During autohesion, segments of the chainlike molecules making up the thermoplastic diffuse across the ply interface. Maximizing molecular diffusion insures a strong bond at the ply interfaces. The compaction mechanism applies a pressure at the ply interface to compact the individual layers of prepreg into intimate contact.

When two surfaces of thermoplastic prepreg tow come into intimate contact, the temperature of the thermoplastic on the mating surfaces should be above its processing temperature. Above this temperature, no crystalline structure will exist in the resin rich surface of the prepreg and the thermoplastic has maximum flow capabilities. At this point consolidation (bonding and compaction) takes place. The extent of molecular diffusion during bonding and the ability of the molten thermoplastic to flow under the force of compaction coincides with the tow feed rate. The desired crystallinity is achieved by cooling from the processing temperature to below the glass transition temperature at an appropriate rate.

ICI Fiberite suggests [8] that the processing temperature of PEEK fall within the range of 380 °C - 400 °C (720 °F - 750 °F). The manufacturer also specifies a cool down rate between 10 °C and 700 °C/min (18 °F and 1260 °F/min) to a temperature of 150 °C (300 °F), the approximate glass transition temperature of PEEK. There is little variation in crystallinity in this cooling range resulting in optimum matrix properties.

## PROCESS DEFECTS

Defects at the ply interface result from improper consolidation of the thermoplastic prepreg. Voids, resin rich areas, resin starved areas, and delaminations are the most likely defects caused by process deficiencies. Defects can be minimized by adjustment of heat and compaction pressure parameters at a given tow feed rate. The distribution of heat must be adjusted so that both surfaces of the consolidating ply interface reach processing temperature at the area under pressure. Crystallization defects may occur uniformly or may vary throughout the the manufactured composite part. If the cooling rate due to free convection falls below the manufacturers specified rate, additional heat must be extracted from the process using forced convection. Thermal cycling during the application of consecutive plies may also cause crystallization defects depending on the range of temperature swings.

Voids and resin rich/resin starved areas may occur as defects at the ply interface. Voids are air deposits that form at the ply interface and between tow widths due to poor thermoplastic flow. Voids produce stress concentrations in a composite part. Resin rich defects appear at the ply interface as areas containing excess thermoplastic while resin starved defects appear as areas lacking thermoplastic at the ply interface. These defects are also caused by poor thermoplastic flow. Both resin rich and resin starved defects in a composite will result in uneven fiber distribution.

Delaminations occur when insufficient flow dampens the autohesion process. In this situation, there is no molecular diffusion across the ply interface resulting in an unbonded segment of tow. A delamination may produce a very large void or a delamination may exist without a discernible void (a "kissing delamination"). There also exists the possibility of poor molecular diffusion, in which case the full interlaminar strength of the composite is not developed.



Insufficient and excessive crystalline structure are other process defects. When processed correctly, proper crystallinity will be achieved and optimum composite properties will result. However, in the case of PEEK APC-2 [8], if the cooling rate falls below 10 °C/min the composite will have excess crystallinity. Excess crystallinity results in a loss of matrix toughness. On the other hand, if this cooling rate exceeds 700 °C/min, the thermoplastics spherulitic growth will not achieve completion. This results in reduced stiffness and resistance to toxic environments.

## TEST TECHNIQUES

### Optical Testing Techniques

There are several microscopy techniques available to assess defects in composite materials both visually and quantitatively. Magnified images of composite cross sections can be used to identify fracture surfaces, voids and resin rich/resin starved areas and their sizes relative to the surroundings. These images can be further analyzed using techniques that can quantify the relative size of defects.

Fracture analysis, a technique utilizing a scanning electron microscope, is used to analyze fracture surfaces [9]. Including its ability to view areas at extremely high magnifications (i.e. 10,000 X), scanning electron microscopy is capable of viewing a non-planar fracture surface with good resolution of all depths of the fracture. Fracture analysis can be used to determine the mechanism of failure, for example, to determine if the mechanical failure of a composite was due to lack of adhesion (resin to fiber bond), or cohesion (resin to resin bond). SEM can also be used to analyze the crystalline structure of a thermoplastic [5].

Micrographs are a viable method of evaluating consolidation [10]. The production of photo micrographs through conventional optical microscopy (i.e. 200 X) is a relatively simple procedure and can be used to identify consolidation defects occurring at the ply interface. A micrograph of sufficient magnification can be used to distinguish between individual plies, showing resin rich/resin poor areas and interply voids. Figure 3 depicts a micrograph from the polished cross section of a prepreg tow constructed composite.

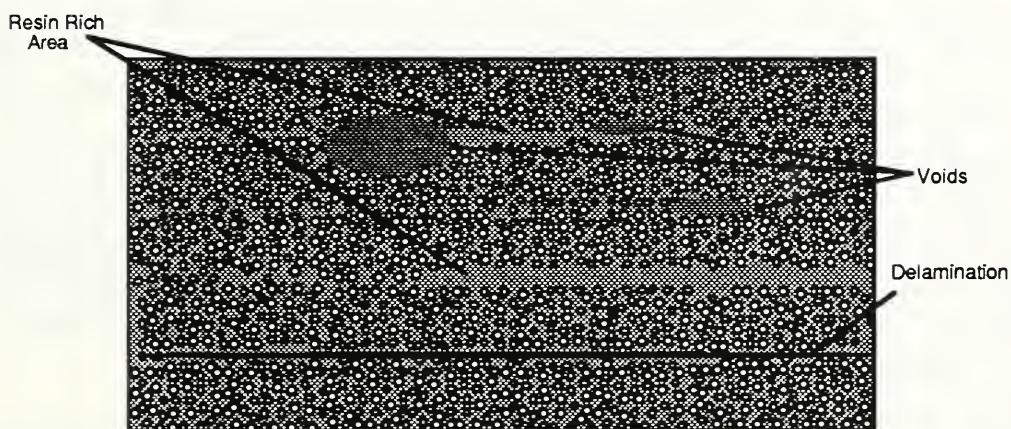


Figure 3 Depicted Micrograph

Quantitative optical microscopy techniques [11] determine fiber, resin, and void contents in carbon reinforced composites by measuring reflected light from a series of microscopic

areas. Reflected light from the polished cross section of a composite are digitized into a video pixel image. Image analysis is then used to assess defects through areal analysis. Defects can be assessed locally or multiple sections can be integrated for a global assessment of the composite.

Optical numeric volume fraction analysis [12] is a variation of the standard quantitative optical microscopy technique. This image analysis technique is capable of quantifying the fiber volume fraction of a highly polished cross sectional sample of a continuous fiber composite by automatically counting the fiber ends per unit area in digitized microscope images. The fiber volume is then computed using a specified value of the reinforcement fiber cross sectional area. This technique is more useful in assessing fiber volume if fiber cross sections are too small to obtain the resolution needed with the more conventional technique using image analysis. Void volume fractions may be obtained from the same digitized images by conventional areal analysis with reasonable accuracy since void dimensions are usually large enough to obtain reasonable resolutions.

## **Physical Testing Techniques**

Physical test techniques exist which have the ability to quantify both consolidation and crystallinity defects. Matrix removal can quantify fiber volume fraction. Density measurement techniques are capable of quantifying void content in composites. Differential Scanning Calorimetry (DSC) can measure the percentage of crystallinity in a thermoplastic matrix.

Two physical tests often used for determining the fiber volume fraction of plastic matrix composites are the ASTM Standard C 613 "Resin Content of Composites by Solvent Extraction" and ASTM Standard D 3171 "Fiber Content of Resin Matrix Composites by Matrix Digestion" [13]. Determinations of volume fraction through matrix removal by either solvent or acid digestion methods involve boiling the composite in toxic chemicals for a period of hours followed by washing and drying the fiber mass prior to weighing. These methods are undesirable for use with thermoplastic matrices since no suitable solvent is available which can remove the matrix without attacking the fibers [12].

Physical tests commonly used to determine the void content of plastic matrix composites are found in ASTM Standard D 2734 "Void Content of Reinforced Plastics" [13]. The densities of the resin, the reinforcement, and the composite are measured separately. The resin content is measured and a theoretical composite density is calculated. This is compared to the measured composite density. The two methods that are available for measuring the actual composite density are ASTM Standard D792 "Test Methods for Specific Gravity (Relative Density) and Density of Plastics by Displacement", and ASTM standard D 1505 "Test Method for Density of Plastics by the Density Gradient Technique" [13]. The accuracy of these techniques depends on the size and distribution of voids in the composite. Large voids with non-uniform distribution may cause considerable variation in results among specimens.

Differential Scanning Calorimetry (DSC) is used to measure the crystallinity of a composite [6]. The technique measures energy absorbed (endotherm) or released (exotherm) by a sample during a process cycle. When an already processed thermoplastic sample is reheated to melt temperature from its fabricated state, a reorganization of the crystalline structure occurs, and energy is released. The ratio of the heat given off by the specimen to the total heat required to melt the specimen provides an indication of the



specimens crystallinity.

## Mechanical Testing Techniques

The ply interface is the primary area of interest when consolidating thermoplastic prepregs. Mechanical tests are discussed here in regards to their ability to assess the ply interface of a consolidated composite. These mechanical tests are comprised of both ASTM Standard test techniques and nonstandard test techniques [13] [14]. Five static tests are considered to assess the consolidation of the simple unidirectional laminate composites. Tests include tensile, compression, interlaminar shear, fracture toughness and flexure tests [15]. Test specimens, in the form of NOL rings, will be fabricated from hoop wound cylinders. A NOL ring is a parallel filament or tape wound hoop test specimen developed by the Naval Ordnance Laboratory (NOL), now the Naval Surface Weapons Laboratory. Procedures and dimensions for the fabrication of NOL rings can be found in ASTM Standard D 2291 "Fabrication of ring test specimens for Glass - Resin Composites". The dimension ranges for NOL rings are shown in figure 4. Dimensions vary within the recommended ranges depending on the mechanical test being performed. Various fixturing and loading schemes are available for performing mechanical tests using NOL rings.

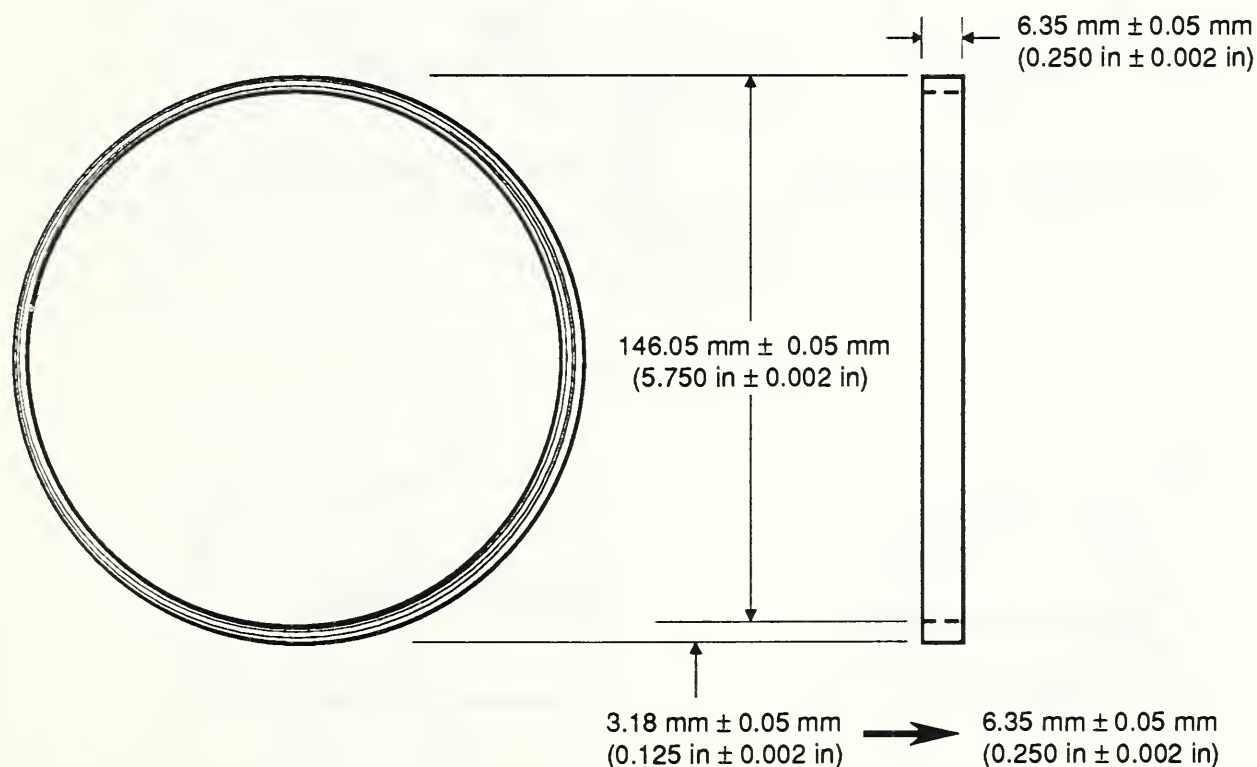


Figure 4 NOL Ring Specimen Dimensions

Tensile tests can be used to test a NOL ring specimen both circumferentially and axially. One circumferential test is the ASTM standard D 2290 "Test Method for Apparent Tensile Strength of Ring or Tubular Plastics and Reinforced Plastics by Split Disk Method" (figure 5a). There is also a non standard hydrostatic pressure circumferential test (figure 5b). Results obtained from these tests are dominated by the tensile strength of the fibers. The ply interface properties have little affect on the circumferential tensile strength of the composite, although the strength of the fiber/matrix bond may cause a change in appearance of the fracture surface. The interlaminar tensile tests (figure 5, c & d) are dominated by the tensile properties of the matrix material. The method in figure 5c loads the matrix perpendicular to the bond surface. This loading configuration does not stress a specimen in the plane of the ply interface. However, the interlaminar tensile test in figure 5d applies a tensile force which directly stresses the ply interface.

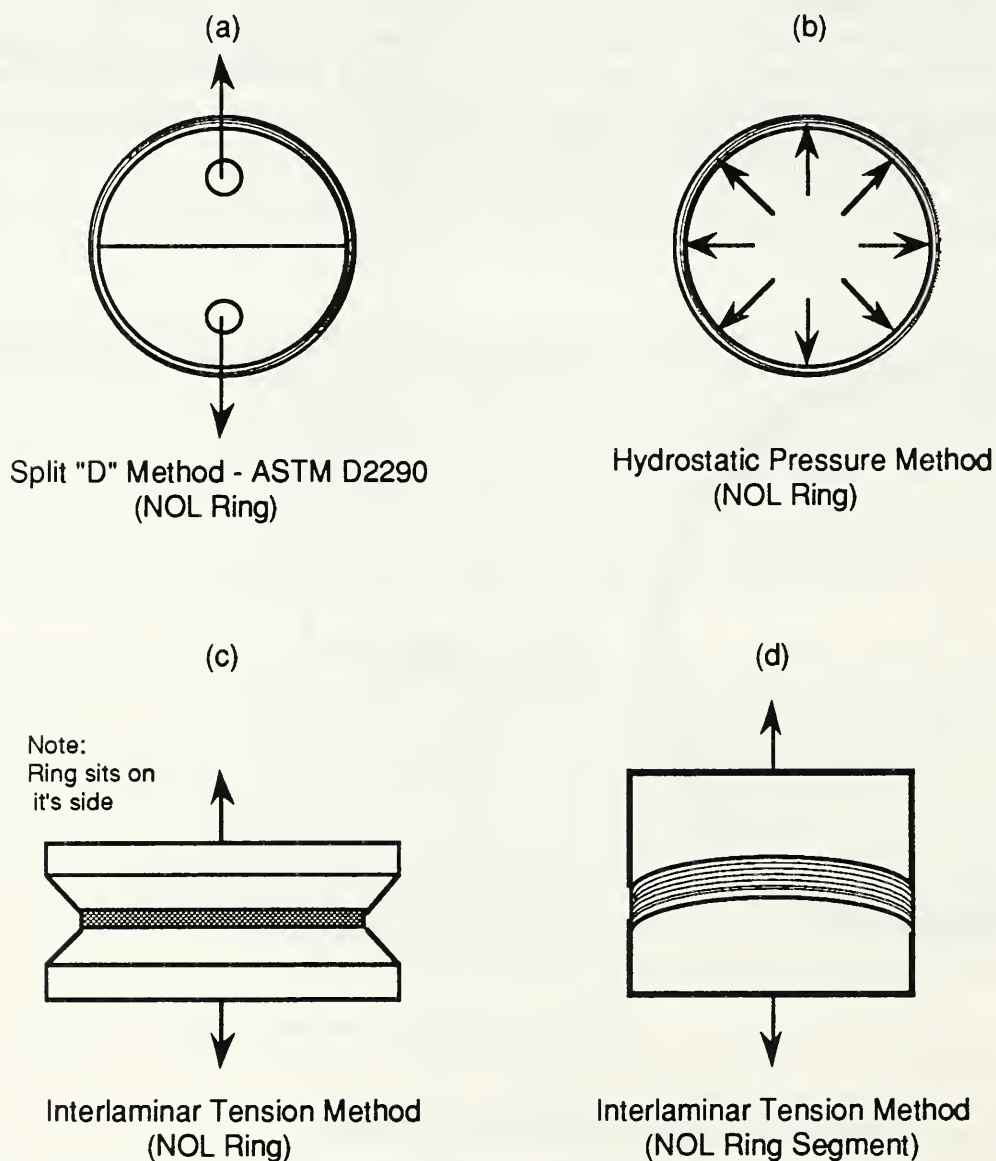


Figure 5 Tension Testing Techniques



Compression test configurations using NOL-rings are shown in figure 6. All five of these techniques involve the same mechanism of failure. The compressive strength and therefore the compression test results of laminate composites are governed by the stability of the fibers. The presence of defects adversely affects the stability of the fibers. During compression testing early interlaminar shear failure occurs due to defects, which can lead to fiber buckling rather than compressive failure of the fibers. Therefore, compression strength is very dependent on interlaminar defects within a composite material.

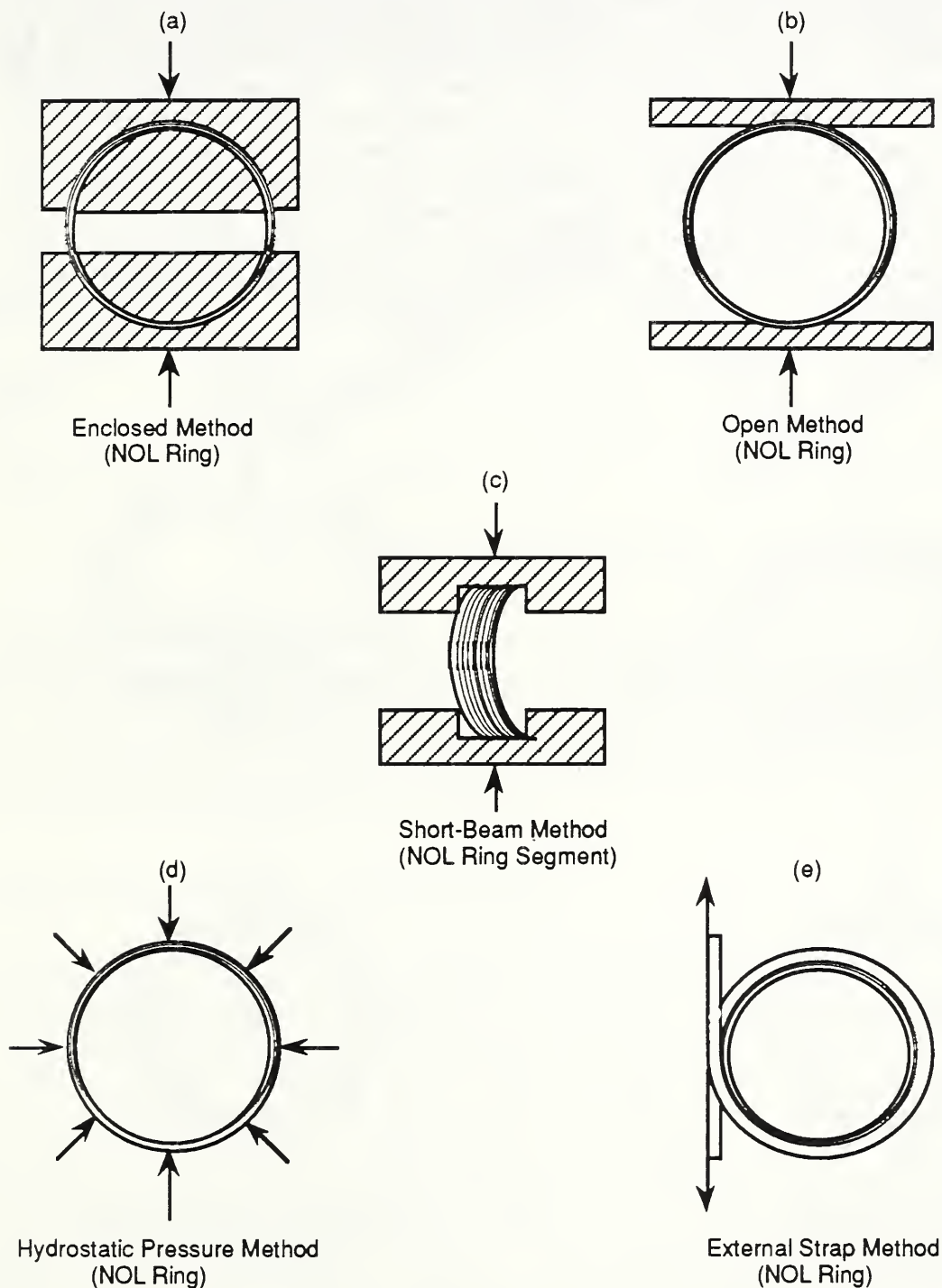


Figure 6 Compression Testing Techniques

Interlaminar shear tests using NOL rings are shown in figure 7. Specimens undergo a loading which attempts to slide individual (plies) past one another. The failure mechanisms of interlaminar shear tests involve the separation of the plane interface between consolidated plies of the laminate. If defects are present at the ply interface, the force required to cause separation at the ply interface of the composite will decrease. A frequent problem existing in shear testing techniques is caused by a high resistance to shear failure. In these cases, the actual failure is via tensile or compressive stresses away from the region of shear. In the ASTM Standard D 2344 "Apparent Interlaminar Shear Strength of Parallel Fiber Composites by Short-Beam Method", a segment of a NOL ring is tested (figure 7a). As the load is applied, the top surface is stressed in compression while the bottom surface is stressed in tension. Due to the tension and compression in adjacent surfaces near the mid-plane, a nearly pure shear stress exists at these plies. Specimen dimensions are designed so that failure is most probable at the mid-plane ply interface rather than at the surface fibers, however, as indicated above this is not often the case. The interlaminar axial shear test (figure 7b) subjects the ply interface to a shear stress applied perpendicular to the fiber direction. Interlaminar shear test techniques using the Split "D" Method and the Hydrostatic pressure method (figure 7, c & d) subject the ply interface of a NOL ring to shear. Notches machined into the specimens inhibit failure by shear in the direction parallel to the reinforcing fibers.

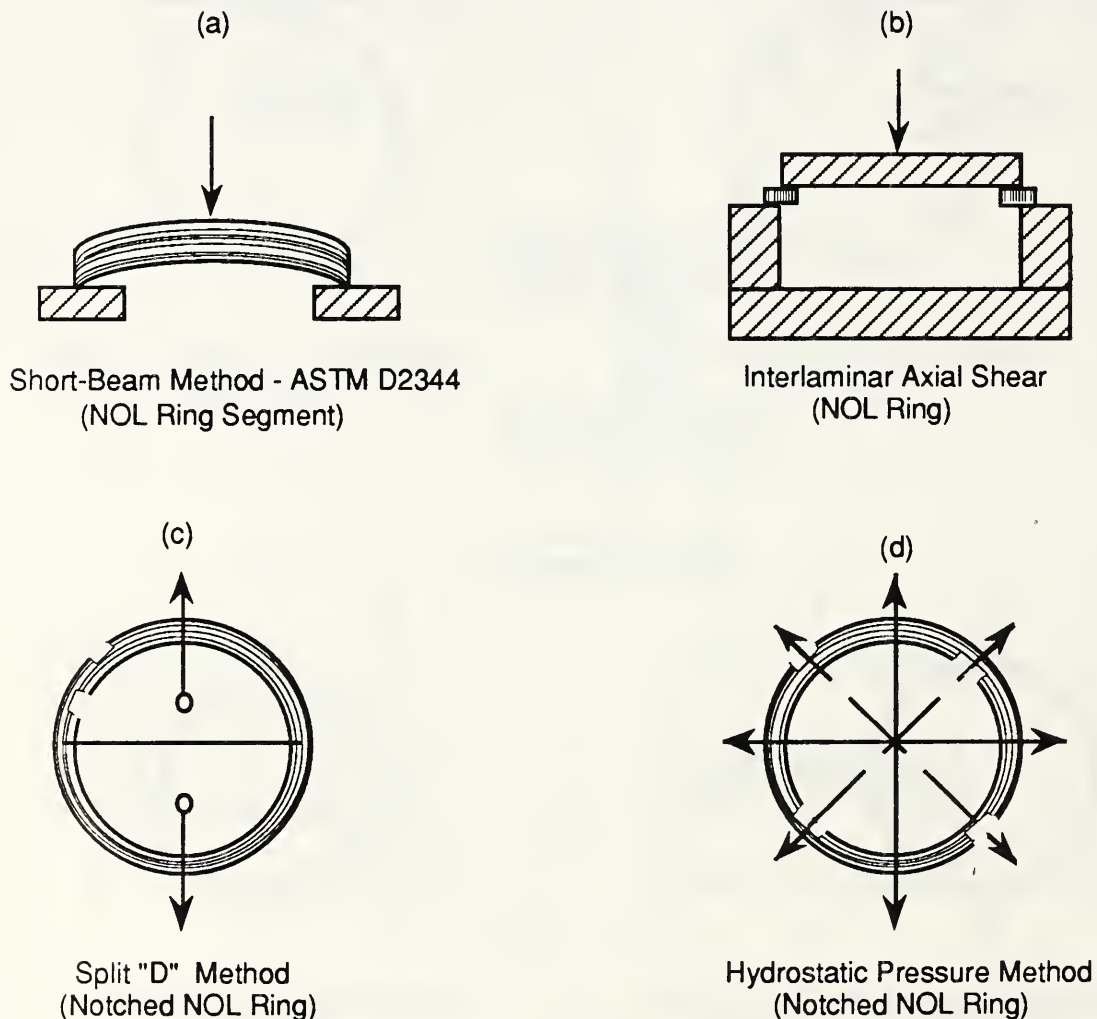


Figure 7 Interlaminar Shear Testing Techniques

Fracture toughness test methods are used to determine the strain energy release rates of a composite material. Materials in general are assumed to fracture in one of three modes (Figure 8), Mode I - the opening mode, Mode II - the shearing mode, or mode III - the tearing mode. Modes I and II would best assess the ply interface in the direction of the fibers. Fracture toughness testing compares the load being applied to the test sample versus the developing crack length. Changes in the load at different crack lengths could correspond to defects at the ply interface of the propagating crack. Although figure 8 depicts the fracture modes using beam type specimens, similar specimens could be fabricated from NOL ring segments. However, ring segments would require modifications to standard test techniques.

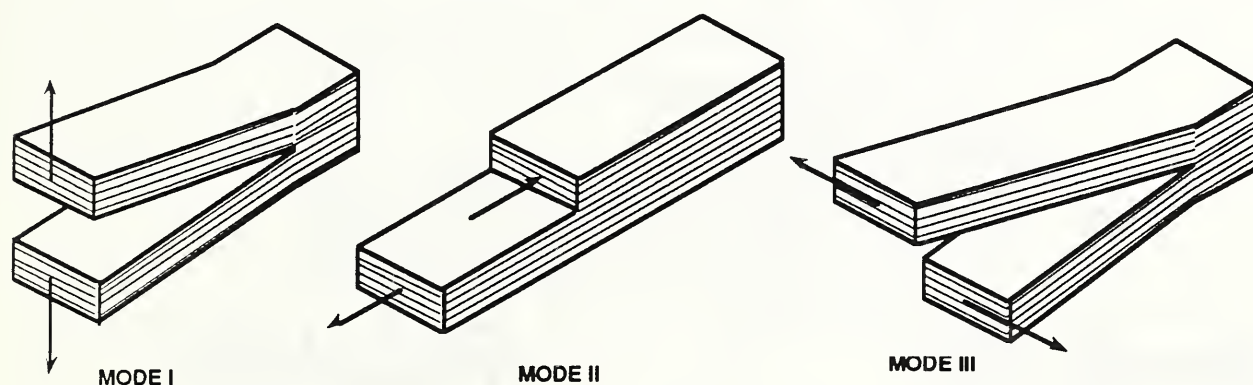


Figure 8 Fracture Toughness Testing

NOL ring flexure tests (Figure 9) are used to measure the bending strength of a composite. When in bending, the composite is in tension along one surface and compression along the opposite surface. As in the short beam shear test, interlaminar shear is developed at the ply interfaces. However, the axis of pure shear is distributed over a large area and is usually not the failure mechanism in this test. This test usually produces failure due to tension and/or compression at the surfaces of the specimen.

### Non Destructive Evaluation Test Techniques

Non destructive evaluation (NDE) techniques [16][17][18][19][20] are used to test for defects without destroying the composite specimen under inspection. Signals capable of penetrating a composite specimen are used to both locate and quantify defects. Utilization of NDE techniques to their full capabilities requires that signals be correlated to defects of known sizes. However, signals can also be used to simply locate defects. Techniques included in the following discussion are ultrasonics, X-radiography, acoustic emission, acousto ultrasonics, thermography and electronic shearography. The capabilities and limitations these various NDE techniques be discussed in context to laminated composites (ie. PEEK APC-2 composites fabricated from prepreg tape). These techniques also will be discussed regarding their ability to be used for off-line detection of defects and an on-line inspection system for the composites workstation. Futuristic goals of an on-line inspection system are to detect process defects shortly after they are induced into a part being produced. The process could then backtrack to the defected area and reapply heat and pressure to eliminate the defects.



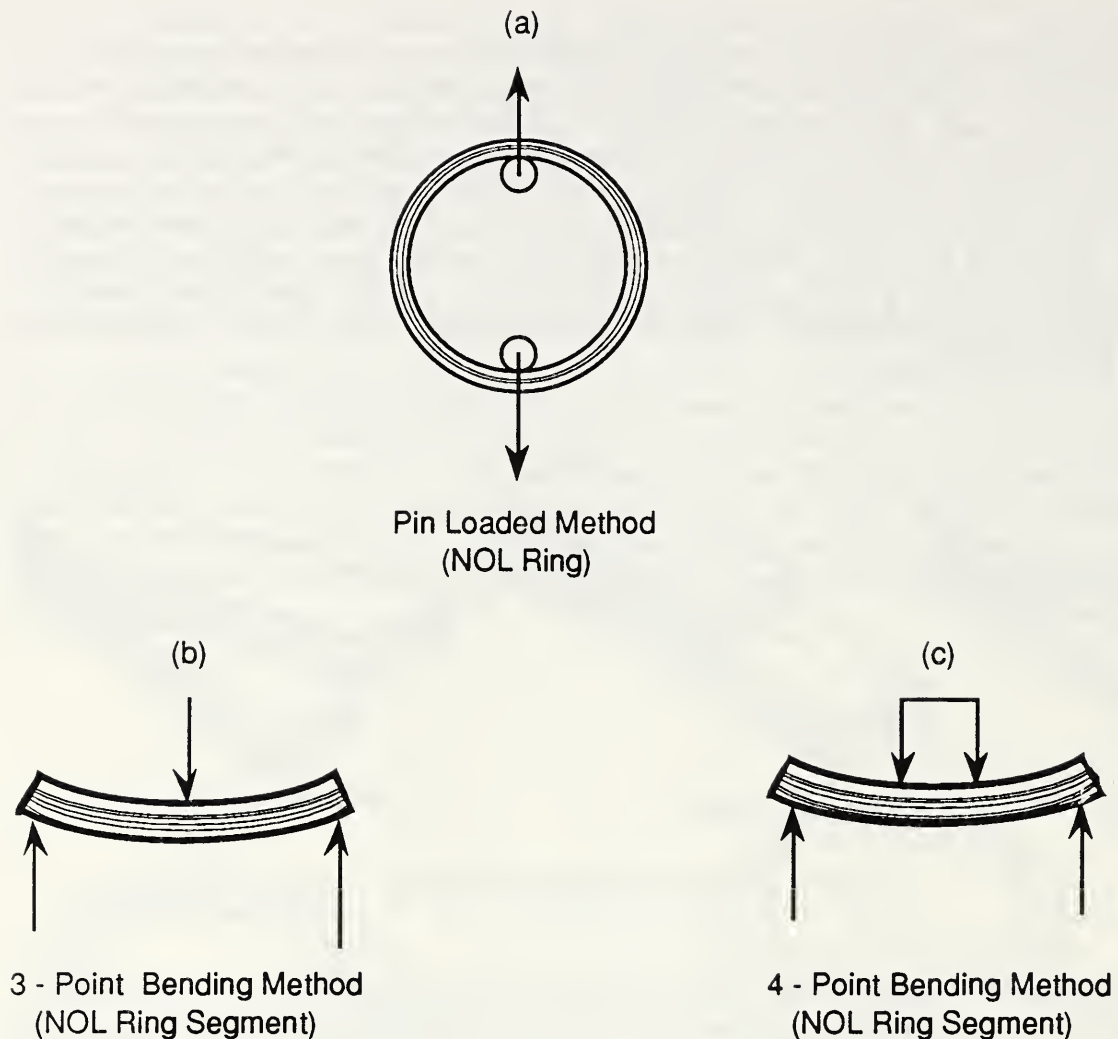


Figure 9 Flexure Testing Techniques

Ultrasonic through transmission and ultrasonic pulse-echo NDE techniques are used to assess laminated composites. These two ultrasonic techniques are often used in automated inspection applications. The through transmission ultrasonic technique uses two transducers to measure the signal strength of a pulse of ultrasonic energy transmitted through a composite. One transducer emits the signal into the composite while the second transducer, located on the opposite side of the composite, receives the signal. Defects in the composite will cause increased signal attenuation. Results of through transmission tests are usually displayed as a C-scan. The through transmission technique is depicted in figure 10.

The pulse-echo ultrasonic technique detects defects by monitoring the time of flight (T.O.F.) and/or the strength of the returning signal. In this case, the pulse of ultrasonic energy is both transmitted and received by a single transducer. Voids and delaminations will cause the reflection of waves at the defect site rather than at the back surface. The reduced T.O.F. and relative signal strengths of these waves indicates a defect. Resin rich defects will delay the back surface reflection due to the increased thickness of the composite in the area of excess matrix material. Figure 11 depicts the pulse echo technique along



with an A-scan oscilloscope display. The horizontal base line indicates elapsed time while the heights of vertical deflections represent intensity of echoes caused by underlaying defects. C-scan displays can also be produced by gating a desired region of several A-scans. The gated vertical deflections of each A scan are plotted in a planar representation of the sample being scanned. Often, these C-scan are represented using color scaled plots. The third method of presenting an ultrasonic scan is the B-scan. A B-scan, usually displayed on a storage oscilloscope, simultaneously displays the essential features of several A-scans, such that the horizontal deflection is proportional to the transducer position and the vertical deflection is proportional to time (or depth through the sample).

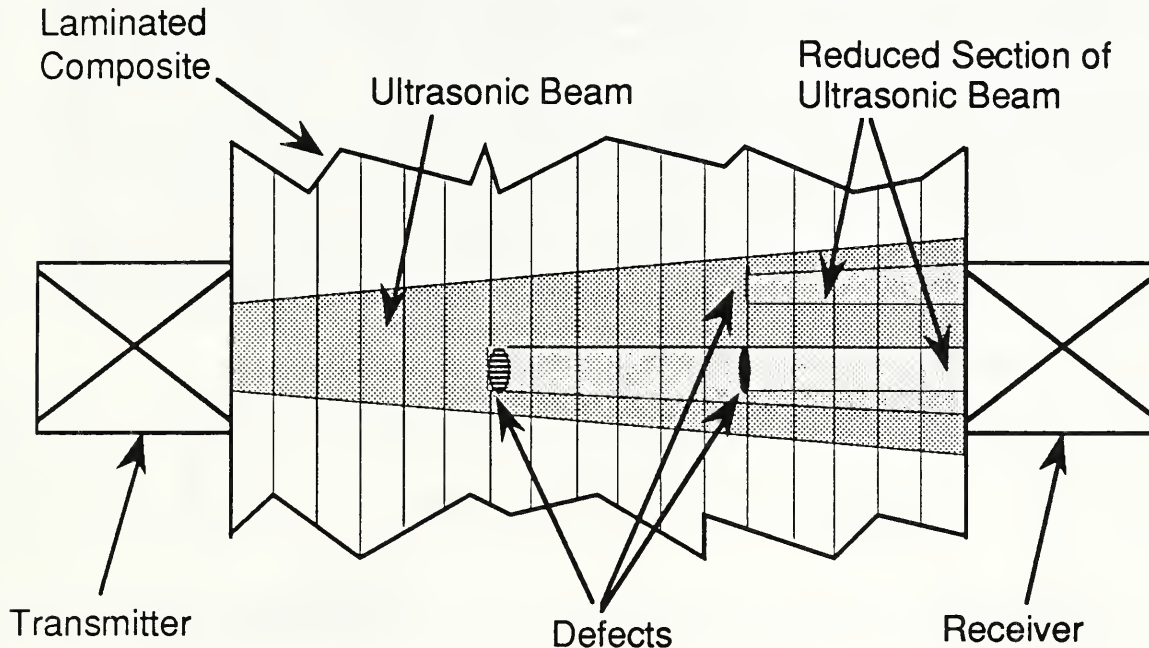


Figure 10 Depiction of Through Transmission

Unlike the through transmission technique, the pulse echo technique enables defects at multiple depths to be distinguished from one another. The pulse echo technique is also advantageous in that it offers increased sensitivity to foreign material inclusions. Kissing delaminations can be detected and void content can be detected in the  $\pm 1\%$  by volume range. One limitation of ultrasonic techniques occurs when a defect of sufficient size reflects a large fraction of the incident sound wave. In this case only a small proportion will propagate beyond the defect. Any additional defects may not be detected.

The ultrasonic through transmission and the ultrasonic pulse-echo techniques both require a couplant to pass the signal from the transducer to the sample. Couplants eliminate acoustical impedance at the interface due to air. Submersing the composite sample in a water bath not only provides an excellent couplant, but also allows the transducer to scan the sample without direct contact (also called the non contact or submersion method). The use of gels and special polymers (also called wet contact placement method and dry contact placement method respectively) are similarly used to minimize the impedance between the composite sample and transducer(s). Water squitters and wheel transducers constructed of special polymers are often used as couplants in automated inspection applications. Other methods of coupling an ultrasonic beam to a test sample include passing the signal through

air (air couplant) and laser induced ultrasonics. These methods have not been studied in detail for composite applications.

Future expectations of an on-line NDE inspection system using conventional ultrasonic inspection techniques are limited due to coupling requirements. Foreign matter cannot be introduced into a thermoplastic composite part as it is being consolidated. Therefore the use of water or gels is prohibited in this application. Since the composite is being wrapped around a mandrel to form a part, the part can only be scanned from one side. These limitations restrict a conventional on-line ultrasonic inspection system to a dry contact pulse echo system. This system in itself is very limited due to signal losses. An on-line inspection system incorporated into the Composites Workstation will be required to inspect the latest three plies of a part being produced. Typically, defects in these near surface regions are covered up by the initial scan pulse. Alternatives to these ultrasonic approaches include air coupled and laser ultrasonics. Both techniques have the advantages of being dry and remote, however, have the disadvantage of being in a developmental stage. Air coupled ultrasonics uses air as the coupling medium for both the transmitted and received signals. Laser ultrasonics uses one laser source to act as a transducer, generating sound when striking a surface, and a second laser source to measure the deflection of a surface after the sound from the first laser has traveled.

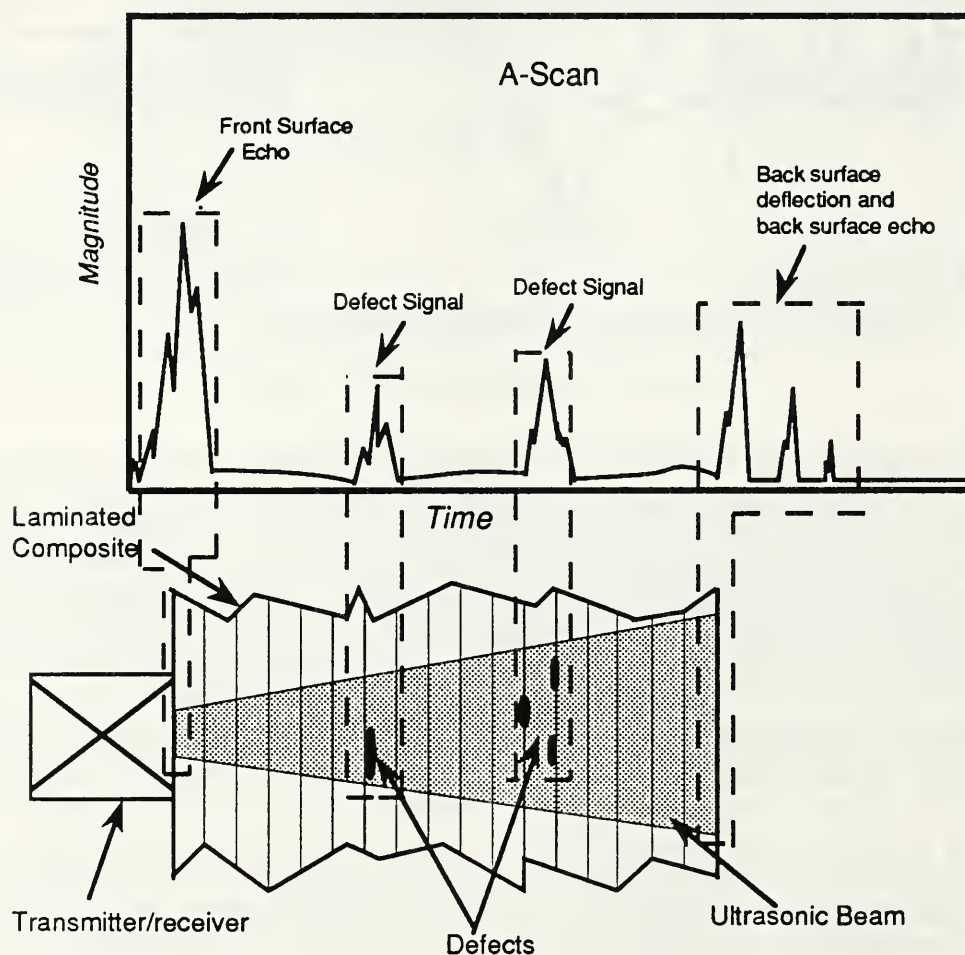


Figure 11 Depiction of Pulse-Echo Transmission



X-radiography and neutron radiography techniques subject the composite material under investigation to penetrating irradiation. Density and thickness variations within the specimen are detected by varying degrees of radiation penetration which are recorded on film. Defects can be measured in real time using fluoroscopes. X-ray diffraction is a radiographic technique which utilizes the diffractive tendencies rather than the penetrating ability of the radiation. The X-ray diffraction radiographic technique is particularly useful to quantify the fiber content and crystallinity [6] in a composite.

The X-radiography technique is capable of detecting void content in the  $\pm 10\%$  by volume range, while the neutron radiography technique is capable of detecting void content in the  $\pm 1\%$  by volume range. Resin rich/resin starved areas are often detected using radiography. One type of defect that can not be easily detected using radiographic techniques is "kissing delaminations". Kissing delaminations can be detected to a certain extent if the damaged region is accessible to penetrant-enhanced radiography. Two other limitations of radiographic techniques are the production of radiation which is a safety problem and the fact that the composite under inspection must be accessible from both sides. These two limitations eliminate radiography as a possible on-line NDE inspection system for the Composites Workstation.

The acoustic emission inspection technique detects defects in a composite specimen by interpreting ultrasonic signals in the form of elastic stress waves which are generated in solids as the result of the mechanical application of stress. The waves are produced by the rapid release of energy within the material. In fiber reinforced composites the stress waves are produced by cracking of the matrix, debonding of the matrix from the fibers, lamination separation, fiber pull-out or breakage of the fibers.

Acoustic emission is not applicable as an on line inspection system to detect voids and resin rich/resin starved areas, probable initiators of the above failure mechanisms, since the stress being applied degrades the structure being analyzed. However, there is the possibility of monitoring stress waves from defects that are created when cooling thermoplastic parts after their high temperature processing. Another implication is the fact that composite materials have a complex set of possible deformation and failure mechanisms and therefore have complicated acoustic emission signatures. In detecting delaminations and fiber misalignments the method requires an extensive database relating various signals to type of defect, degree of defect, and specimen geometry.

Acousto-Ultrasonics combines some aspects of acoustic emission with ultrasonic simulation of stress waves. Acousto-Ultrasonics uses two transducers on the same side of the material. One transducer produces an ultrasonic signal to simulate stress waves in the material, while the other receives the resulting ultrasonic signal produced by the variations in the composites mechanical properties as in acoustic emission. Substantial material degradation is less likely than in using the acoustic emission technique, however, complex signatures are produced as in the acoustic emission technique. The transducers must also remain in contact with the material and it's surface geometry must be known. This eliminates the techniques use as an on-line inspection technique.

Thermography is a technique used to locate defects by monitoring thermal gradients appearing on the surface of a composite part. These thermal gradients can be used to estimate both the location and the characteristics of defects. Thermography has two modes of operation, active and passive. The active mode of thermal inspection subjects a composite to a stress (mechanical or vibrational). Under this cyclic loading heat is generated in regions of damage through various mechanisms (ie. matrix cracking). These

mechanisms generate thermal gradients on the composite part surface. The passive mode of thermal inspection involves the application of a local or general source of heat followed by observation of the subsequent temperature gradients produced by underlying defects. The thermal gradients can be recorded using an infra-red sensitive camera. The composite workstation's hot-air heating system makes passive thermal inspection the most likely candidate for an on-line inspection system. Thermography can detect defects at ply interfaces, however, since the part being inspected on-line can only be accessed from one side, the depth of inspection is limited. Thermography, a non-contact technique, is a possible system for use as an on-line inspection system provided that the system being used can identify defects as far as three plies below the surface.

Shearography is a technique that detects defects by employing laser interferometry to a stressed composite part. This interferometric technique provides whole field observation of small surface strains caused by underlying defects. The technique is most effective when a composite part is stressed under a vacuum, however, a part could be stressed thermally with less accurate results. Shearography is capable of detecting defects at ply interfaces and uses air as a coupling medium. The technique could be limited for use in the composites workstation because of its size and depth limitations to detect defects.

## **A TEST METHODOLOGY**

### **Off-Line Inspection**

A test methodology (figure 12) to measure the quality of composite parts produced by the composites workstation was chosen based on a trade-off analysis of all considered testing techniques. Test techniques chosen for implementation into the test methodology include low magnification microscopy, density measurements, differential scanning calorimetry, short beam shear testing and ultrasonic inspection. The trade-off analysis also includes discussions for alternative techniques. Possible techniques discussed for use as an on-line inspection system include ultrasonics, thermography, and electronic shearography.

The physical, mechanical, and non-destructive test techniques were evaluated based their capability to locate, identify, and quantify defects at the consolidated ply interface. Optical test techniques were evaluated on their ability to identify and estimate general sizes and distributions of defects. Physical test techniques were evaluated on their potential to quantify defects. Mechanical test techniques were evaluated on their potential to be used to assess the interlaminar strength of the consolidated composite. Non-destructive test techniques were evaluated on their potential implementation for off-line inspection of already fabricated parts and for on-line inspection feedback of the manufacturing process. Other factors taken into account when evaluating test techniques were the availability and cost of equipment and the complexity of the test technique.

Cross sections of composite specimens will be evaluated using conventional microscopy techniques. Photo micrographs produced will be used to both identify and assess the general size and distribution of defects. Another physical test technique considered for identifying the ply interface defects was Scanning Electron Microscopy. Equipment used in this technique, although capable of identifying defects, is designed for assessing a material at much higher magnifications specifically for applications such as analysis of fracture surfaces and crystalline structure. These are not immediate concerns to processing efforts but may be considered in future efforts.



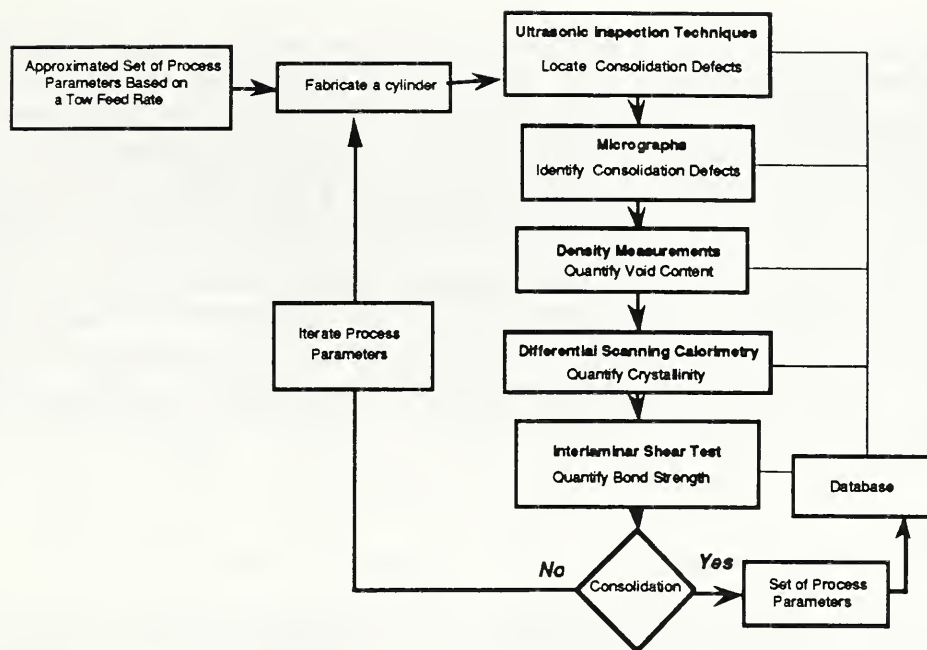


Figure 12 Test Methodology

The density measurements specified in ASTM Standard D 2734 appear to be the most viable techniques for quantifying void content. Based on the availability of equipment and expertise, the first technique used will be the density gradient technique. Other density techniques will be implemented if required. If the need to analyze local void contents arises, quantitative optical microscopy, including standard techniques and optical numerical volume fraction analysis techniques, will be considered. These techniques can also quantify, locally, the percent fiber volume fraction.

Differential Scanning Calorimetry and X-Ray Diffraction will be used in parallel to measure the crystallinity of composite samples produced. DSC uses calorimetric measurements produced from the heat of crystallization to quantify crystallinity while X-ray diffraction quantifies crystallinity using the diffractive properties of the crystalline structure in the material. Both techniques are available.

Based on the various failure mechanisms of tension, compression, fracture toughness, interlaminar shear, and flexure, interlaminar shear tests and fracture toughness tests were determined to be the most applicable mechanical tests. More specifically the Short Beam Shear interlaminar shear test (ASTM Standard D 2344) was chosen as the initial test approach while the fracture toughness test analyzing Mode I or Mode II failure will be implemented as a parallel technique. Short Beam Shear testing will be performed until a degree of consolidation is reached where specimens no longer fail in shear. Fracture toughness testing using ring specimens is not a standard test technique. Future capabilities will allow for the fabrication of specimen flats. Hydrostatic compression testing will also be considered as an alternative technique to measure the strength of the ply interface.

Ultrasonic Inspection techniques were chosen as an off line inspection tool based on their capabilities of locating voids in the  $\pm 1\%$  range, delaminations, and resin rich areas that will appear at the ply interface of the consolidated composite. Either through transmission or

pulse echo ultrasonics can be used as an off-line inspection technique. Coupling systems are also not limited using off-line inspection. Neutron radiography may be considered as an alternative technique because of its capability to locate void content in the  $\pm 1\%$  range and its ability to locate resin rich areas. X-Ray diffraction was considered because of the techniques ability to locate variations in fiber content using it's diffractive properties, however, this is not an immediate concern to our efforts. The other NDE techniques considered for locating and quantifying defects include Acoustic Emission, Acousto Ultrasonics, Thermography, and Electronic Shearography. These techniques are relatively new techniques when compared to the more widely used ultrasonic and radiography techniques. Interpretation of data is difficult because of the complex signatures produced by these techniques. Also, equipment is expensive and not readily available. These techniques will again be considered for implementation of an on-line inspection system in the composites workstation.

## On-Line Inspection

Futuristic goals of the composites workstation are to implement an on-line inspection system. This inspection system would ideally have the capability to monitor composite parts as they are being produced. The non-destructive inspection technique chosen for this application must have the ability to detect defects in the immediate ply layers of a part being produced. When defects are detected, the composite workstation will reiterate it's heating and compaction process over the recently detected area of defects. This system would eliminate part rejections due to process defects.

Techniques which have the most potential to be used as on-line inspection system include air or laser coupled ultrasonics, thermography, and electronic shearography. Initial selection of these techniques was based on their coupling capabilities as well as their defect detecting capabilities. Applicability of these techniques to the composite workstation as an on-line inspection system must be looked at in more detail.

## SUMMARY

A composites workstation, an advanced manufacturing workstation, is being developed in the Automated Manufacturing Research Facility (AMRF) at the the National Institute of Standards and Technology (NIST). This composite workstation will be used to study methods of fabricating high performance, complex shaped, continuous fiber reinforced, thermoplastic composite parts typically used in the aircraft and defense industry. Fabrication will be accomplished using in-situ consolidation and prepreg tow placement techniques via two cooperating robot manipulators.

A test methodology to measure the quality of composite parts fabricated by the composite workstation was presented in this paper. The test methodology utilizes a combination of material testing techniques to measure the consolidation of fabricated parts during implementation of the manufacturing process. Mechanisms of thermoplastic consolidation and the various defects associated with poor consolidation were presented. Several optical, physical, mechanical, and non-destructive testing techniques were discussed concerning their ability to locate, identify and/or quantify these defects. Non-destructive test techniques were also discussed regarding their capabilities to be used as an on-line inspection system. A test methodology was presented as being an effective means to measure the quality of thermoplastic composite parts both off-line and on-line.



## REFERENCES

- <sup>1</sup> R.J. Norcross, W.R. Bunch, and R.D. Kilmer, "AMRF Composite Fabrication Workstation", 3rd International Symposium on Robotics & Manufacturing (ISRM), 1990.
- <sup>2</sup> J.B. Cattanach and F.N. Cogswell, "Processing with Aromatic Polymer Composites", Developments in Reinforced Plastics 5, 1986.
- <sup>3</sup> W.I. Lee and G.S. Springer, "A Model of the Manufacturing Process of Thermoplastic Matrix Composites", Journal of Composite Materials, (November 1987).
- <sup>4</sup> F.N. Cogswell and D.C. Leach, "Processing Science of Continuous Fibre Reinforced Thermoplastic Composites", SAMPE Journal, (May/June 1988).
- <sup>5</sup> J.F. Carpenter, "Thermal Analysis and Crystallization Kinetics of High Temperature Thermoplastics", SAMPE Journal, (January/February 1988).
- <sup>6</sup> H. Motz and J.M. Schultz, "The solidification of PEEK. Part I: Morphology", Journal of thermoplastic Composite Materials, (Vol. 2 - October 1989).
- <sup>7</sup> H. Motz and J.M. Schultz, "The solidification of PEEK. Part II: Kinetics", Journal of thermoplastic Composite Materials, (Vol. 2 - October 1989).
- <sup>8</sup> ICI Fiberite, "Fabricating with aromatic polymer composite, APC-2", Data Sheet #5.
- <sup>9</sup> D. Purslow, "Matrix Fractography of Fiber-Reinforced Thermoplastics", Composites, (Vol. 18 No. 5 November 1985 & Vol. 19 No. 5 September 1988).
- <sup>10</sup> E. Beyeler, W. Phyllips, and S.I. Gucer, "Experimental Investigation of Laser-Assisted Thermoplastic Tape Consolidation", Journal of Thermoplastic Composite Materials, (Vol. 1 - January 1988).
- <sup>11</sup> C.P. Jock, "Quantitative Optical Microscopy Fiber Volume Methods for Composites", Journal of Reinforced Plastics and Composites, (Vol. 5 - April 1986).
- <sup>12</sup> M.C. Waterbury and L.T. Drzal, "Determination of Fiber Volume Fractions by Optical Numeric Volume Fraction Analysis", Journal of Reinforced Plastics and Composites, (Vol. 8 - November 1989).
- <sup>13</sup> "ASTM Standards and Literature References for Composite Materials", (1987).
- <sup>14</sup> N. Fried, "Survey of Test Methods for Parallel Filament Reinforced Plastics", ASTM Symposium on Standards for Filament-Wound Reinforced Plastics, (1969).
- <sup>15</sup> Y.M. Tarnopol'skii and T.Kincis, "Static Test Methods for Composites", (1981).
- <sup>16</sup> D.E.W. Stone and B. Clark, "Non-Destructive Evaluation of Composite Structures - An Overview", Materials and Structures Department, Royal Aircraft Establishment, England.
- <sup>17</sup> J. Summerscales, "Non-Destructive Testing of Fiber-Reinforced Plastics Composites", (Vol. 1 - 1987)
- <sup>18</sup> R.F. Wegman, "Non-Destructive Test Methods for Structural Composites", SAMPE Handbook No. 1, (1989).
- <sup>19</sup> T.S. Jones and H. Berger, "Application of Nondestructive Inspection Methods to Composites", Materials Evaluation, No.47, (April 1989)
- <sup>20</sup> J. Summerscales, "Non-Destructive Testing of Fiber-Reinforced Plastics Composites", (Vol. 2 - 1990).





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☐ DOCUMENT DESCRIBES A COMPUTER PROGRAM; SF-185, FIPS SOFTWARE SUMMARY, IS ATTACHED.

11. ABSTRACT (A 200-WORD OR LESS FACTUAL SUMMARY OF MOST SIGNIFICANT INFORMATION. IF DOCUMENT INCLUDES A SIGNIFICANT BIBLIOGRAPHY OR LITERATURE SURVEY, MENTION IT HERE.)

The Robot Systems Division of the National Institute of Standards and Technology (NIST) requires a test methodology to measure the quality of the composite parts produced by an advanced manufacturing workstation being assembled in the Automated Manufacturing Research Facility (AMRF). The workstation will be used to study methods of fabricating complex shaped, continuous carbon fiber reinforced, thermoplastic composite parts using pre-impregnated tow. Fabrication will be accomplished using in-situ consolidation and tow placement techniques via two cooperating robot manipulators. The test methodology will utilize a combination of material testing techniques to measure the consolidation of fabricated parts during implementation of the manufacturing process. This paper discusses the mechanisms of thermoplastic consolidation and the various defects associated with poor consolidation. Several testing techniques are discussed concerning their ability to locate, identify and/or quantify these defects. This discussion is followed by a trade off analysis of all considered testing techniques in an attempt to determine the most effective test methodology to measure the quality of thermoplastic composite parts.

12. KEY WORDS (6 TO 12 ENTRIES; ALPHABETICAL ORDER; CAPITALIZE ONLY PROPER NAMES; AND SEPARATE KEY WORDS BY SEMICOLONS)	AMRF; Composite Fabrication; Fiber Placement; In-situ Consolidation; Thermoplastic Composites; PEEK APC-2; Composite Testing; Non-Destructive Evaluation (NDE)
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